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13. ABSTRACT (Maximum 200 words)

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REPORT

EPR and Electrical Investigations of Point Defects and Hopping Motion in SiC Heavily Doped with Nitrogen.

(Project SPC-97-4051)

January - May 1997

During this period it was performed the following work and were obtained the following results:

1. The temperature behavior of the donor in 6H SiC n-type samples with uncompensated nitrogen concentration ($N_D - N_A$) from $3 \cdot 10^{18}$ to $1 \cdot 10^{16}$ cm^{-3} in the temperature interval from 20 K to 160 K was investigated by Electron Paramagnetic Resonance (EPR).

2. For the samples with uncompensated nitrogen concentration ($N_D - N_A$) from $3 \cdot 10^{18} cm^{-3}$ to $5 \cdot 10^{17} cm^{-3}$ two donor spectra were resolved in the temperature interval of 20 - 160 K: three ESR spectra associated with nitrogen in three inequivalent positions and additional broad line originated from nonlocalized nitrogen electrons. (Fig.1).

3. For the samples with uncompensated nitrogen concentration in the range of $3 \cdot 10^{17} cm^{-3}$ to $1 \cdot 10^{16} cm^{-3}$ two donor spectra were observed in the temperature interval of 40 -140 K: two EPR spectra associated with nitrogen in two cubic sites and additionall slightly temperature dependent background line of small intensity which was attributed to the residual donor defect presented in SiC samples due to the deviation from the stoichiometry. (Fig.4).

4. Correlation between the temperature behavior of nitrogen in cubic site and concentration of nitrogen in 6H SiC were established. With decreasing of nitrogen concentration from $3 \cdot 10^{18} cm^{-3}$ to $1 \cdot 10^{16} cm^{-3}$ the peak position of temperature dependence of intensities of EPR spectra of nitrogen in cubic sites is shifted to the high temperature from 60 K to 95 K, with the temperature interval being shifted from 4.2 -110 K to 50 - 160K respectively. (Fig.6).

5. The analyse of temperature dependencies of the intensities of nitrogen and additionall EPR lines were performed for the samples with wide range of nitrogen concentration.

6. In the concentration range of $3 \cdot 10^{18}$ - $1 \cdot 10^{18} cm^{-3}$ the values of ionization energies E = 150 meV, 80 meV, 60 meV of nitrogen at cubics, hexagonal sites and of nonlocalized nitrogen electrons state were determined respectively. (Fig.2,3). The value of activation energy E = 16 meV obtained from the low temperature slope of temeperature dependence of intensity of the line from nonlocalized electrons are in a good agreement with those obtained from d.c. conductivity for the samples of same nitrogen

concentration range [1]. This fact was shown that at the concentration of nitrogen higher than $5 \cdot 10^{17} cm^{-3}$ the nonlocalized nitrogen electrons make the principle contribution to the d.c. conductivity in 6H SiC.

7. With decreasing of nitrogen concentration up to $3 \cdot 10^{17} cm^{-3}$ the temperature behavior of EPR spectra of donor states completely changes. One striking result in our experiment is that in the range of low concentration of nitrogen $3 \cdot 10^{17} - 1 \cdot 10^{16} cm^{-3}$, when the concentration of nonlocalized electrons is low one can obtain the value of valley-orbit splitting E = 58 - 52 meV for nitrogen at cubic site from the temperature dependence of the intensity of the EPR spectra of nitrogen in cubic site (Fig.5,7). If the concentration of nonlocalized electrons is too high it should be impossible to observe the valley-orbit transitions between A_1 and E state for nitrogen in cubic site because of it is covered by hopping motion and ionization of the donors.

8. The values of activation energies were obtained from the low temperature slope of temperature dependence of EPR spectra intensity of nitrogen in cubic sites for the samples with concentration range of $5 \cdot 10^{17} cm^{-3}$ - $1 \cdot 10^{16} cm^{-3}$ (Fig.5,7). With decreasing of nitrogen concentration from $5 \cdot 10^{17} cm^{-3}$ to $1 \cdot 10^{16} cm^{-3}$ the value of activation energy was increased from 19 meV to 26 meV respectively and are in a good agreement with those obtained from d.c. conductivity in the same concentration range [1].

   This fact shows that at low concentration of nitrogen the d.c. conductivity was caused by the nitrogen electrons in cubic site.

9. Temperature behavior of ESR spectra of 6H SiC sample with nitrogen concentration of $5 \cdot 10^{17} cm^{-3}$, grown at T = 2500°C, irradiated with neutron flux of $\Phi = 2 \cdot 10^{18} cm^{-2}$ and thereafter annealed at 1400°C which exhibited the red luminescence was investigated in the temperature interval from 4.2 to 160 K.(Fig.8, 9). Analyse of the temperature dependence showed that EPR spectra consist of EPR spectrum associated with nitrogen in two cubic sites and two single lines. One line observed in the low temperature interval form 4.2 K to 40 K and consist of nitrogen in hexagonal site and defect line while another one of small intensity appeared at 60 K and incresed in intensity with increasing the temperature to 160 K. The EPR parameters of low temperature defect were obtained at ν_{EPR} =142 GHz and T = 4.2 K and was intrepreted as a new deep acceptor at E_v + 0.8 eV. The high temperature line could be attributed to the residual donor defect inherent to 6H SiC samples with nitrogen concentration low than $5 \cdot 10^{17} cm^{-3}$.

   The energetically parameters were obtained from temperature dependence of intensity of EPR line of nitrogen in cubic sites (Fig.10). Energy ionization of nitrogen E = 63 meV is in a good agreement with value obtained for the 6H Lely samples with same nitrogen concentration, while the energy activation E = 10 meV obtained from the low temperature slope

of temperature curve is too small compare to those obtained for the Lely samples with the same concentration of nitrogen.

10. To eastablish the nature of native defect presented in the 6H samples with low concentration of nitrogen less than $5 \cdot 10^{17} cm^{-3}$ we investigated the temperature behavior of 6H SiC epilayers (EL) grown at low temperature 1850°C and high velocity $V_g = 0.9 - 1.0$ mm/h with nitrogen concentration of $(N_D - N_A) \approx 7 \cdot 10^{16} cm^{-3}$.

Two EPR spectra were also resolved in 6H SiC EL, but the temperature behavior of EPR spectra is completely different from those for 6H Lely grown samples (Fig.11). One EPR spectra associated with nitrogen in two cubic sites were observed in the temperature interval of 60 -120 K and peaked at 85 K while another one with the intensity of 5 times greater than in 6H SiC Lely grown samples were observed in the temperature interval of 20 -120 K and peaked at 80 K. The energetically characteristics were obtained from the temperature dependences of intensities of EPR spectra. The obtained energy ionization of nitrogen in cubic site E = 112 meV (Fig.12) is not coinced with those obtained for nitrogen in cubic site in 6H Lely samples with nitrogen concentration in the range of $3 \cdot 10^{17} cm^{-3}$ to $1 \cdot 10^{16} cm^{-3}$. The value of energy activation E = 78 meV was obtained from the low temperature slope of nitrogen temperature curve.

The values of energy ionization E = 120 meV and energy activation E = 4.5 meV were obtained for nonidentified EPR line (Fig.13).

There was not yet established what is the donor make the principall contribution to the d.c. conductivity and It is not clear why the energy ionization of nitrogen in 6H EL is not coinced with the value of those obtained for 6H Lely grown samples. Further exploration and comparison EPR data with electrical data would be required.

11. The preliminary investigations of 4H SiC bulk samples grown by sublimation sandwich method (SSM) at T = 1900 -2000°C with the nitrogen concentaration between of $1 \cdot 10^{18} cm^{-3}$ - $7 \cdot 10^{16} cm^{-3}$ were performed. The temperature behavior of EPR spectra of nitrogen in cubic site as a function of impurity concentration in 4H are similar to those obtained for 6H Lely grown samples. Additional donor defects of unidentified nature that observed in 4H samples with the different concentration of nitrogen are under investigations.

12. Eleven 6H, 4H SiC samples for electrical and optical measurements were submitted to Dr. W.C. Mitchel.

References

[1] A. I. Veinger Sov.Phys.Semic. 1, p.14 - 18, 1967.

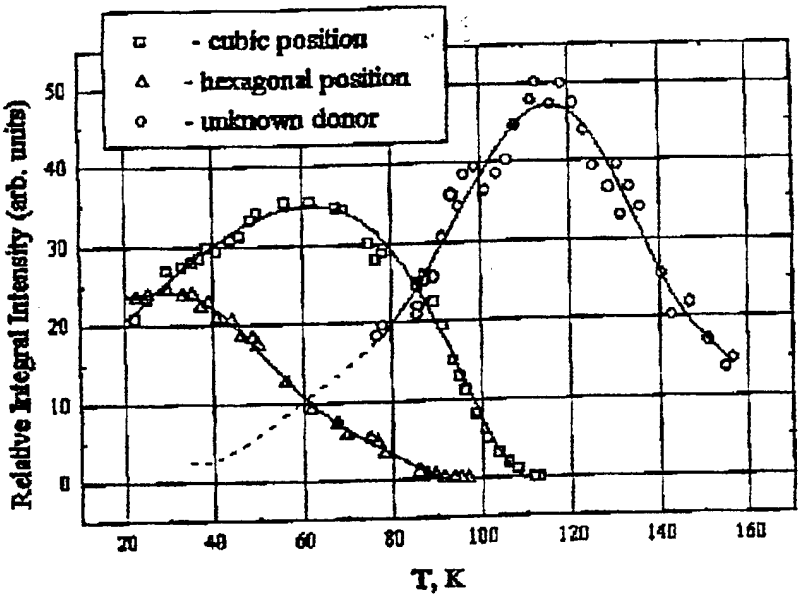

Fig. 1. Temperature dependencies of the relative integral intensities of ESR spectra of bulk 6H SiC with uncompensated nitrogen concentration of $(N_D - N_A) \approx 2 \cdot 10^{18} cm^{-3}$.

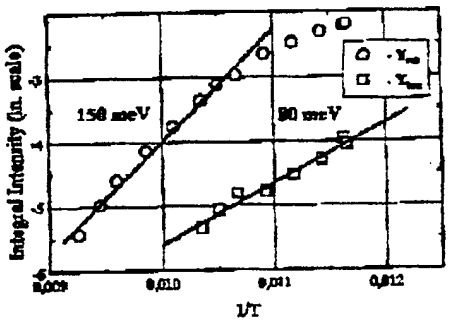

Fig.2. Temperature dependencies of the integral intensities of ESR spectra of nitrogen in cubic sites in bulk 6H SiC with $(N_D - N_A) \approx 2 \cdot 10^{18} cm^{-3}$. The solid lines are fitted by $\exp(\Delta/kT)$ where Δ = 150 meV for cubic sites and 80 meV for hexagonal site.

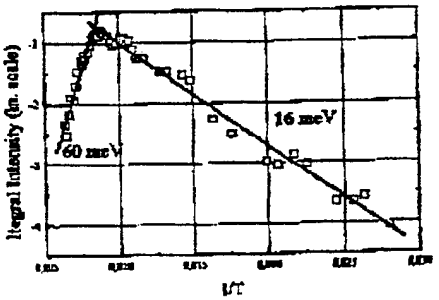

Fig. 3. Temperature dependencies of the integral intensities of ESR spectra of nonlocalized nitrogen electrons in bulk 6H SiC with $(N_D - N_A) \approx 2 \cdot 10^{18} cm^{-3}$. The solid lines are fitted by $\exp(\Delta/kT)$ with Δ = 60 meV and 16 meV.

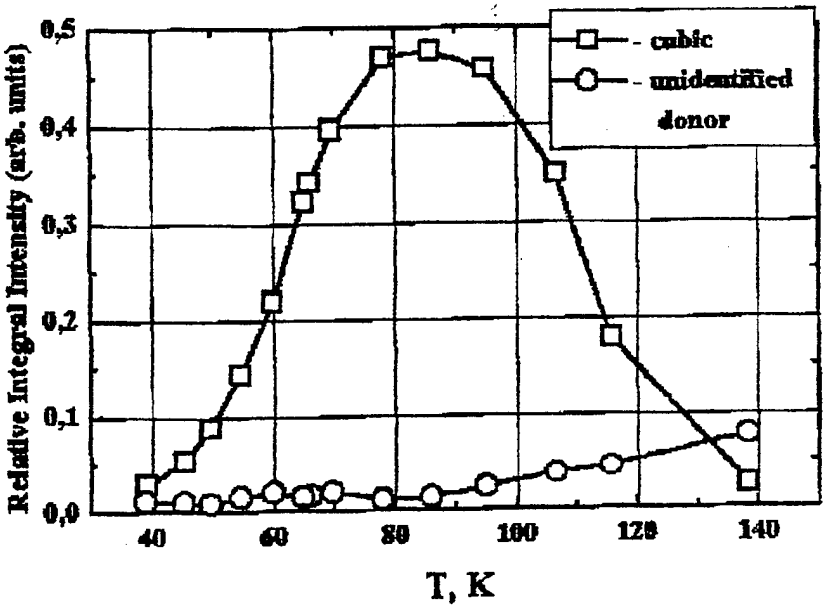

Fig. 4. Temperature dependencies of the relative integral intensities of ESR spectra of bulk 6H SiC with uncompensated nitrogen concentration of $(N_D - N_A) \approx 3 \cdot 10^{17} cm^{-3}$.

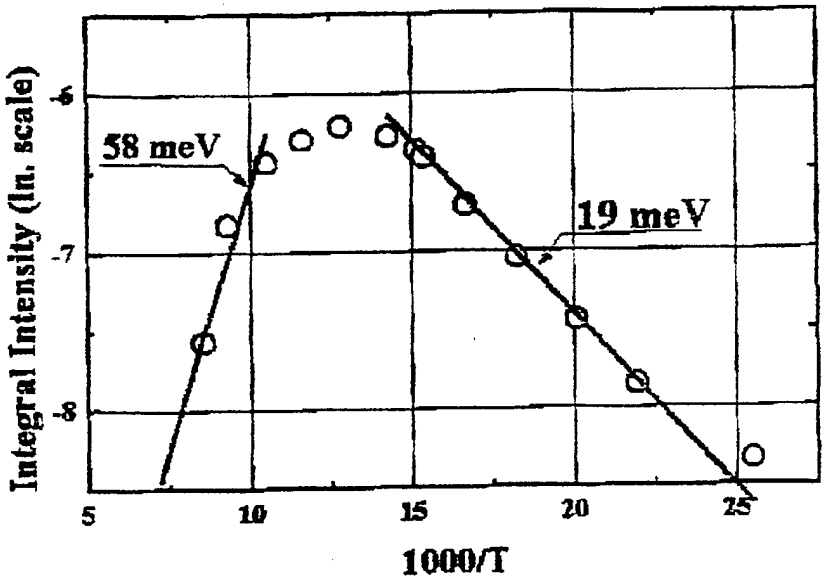

Fig.5. Temperature dependencies of the integral intensities of ESR spectra of nitrogen in cubic sites in bulk 6H SiC with $(N_D - N_A) \approx 3 \cdot 10^{17} cm^{-3}$. The solid lines are fitted by $\exp(\Delta/kT)$ with Δ = 58 meV and 19 meV.

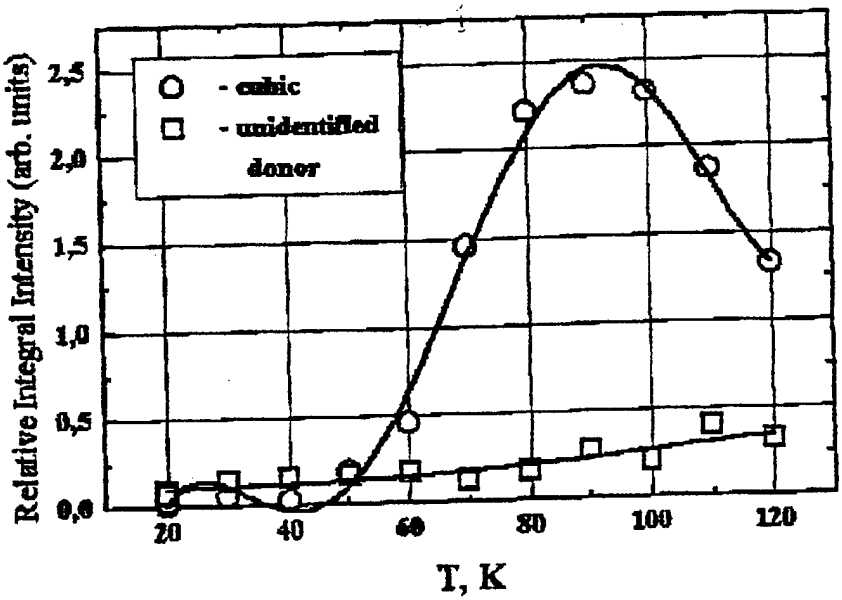

Fig.6. Temperature dependencies of the relative integral intensities of ESR spectra of bulk 6H SiC with uncompensated nitrogen concentration of $(N_D - N_A) \approx 1 \cdot 10^{16}$ cm^{-3}.

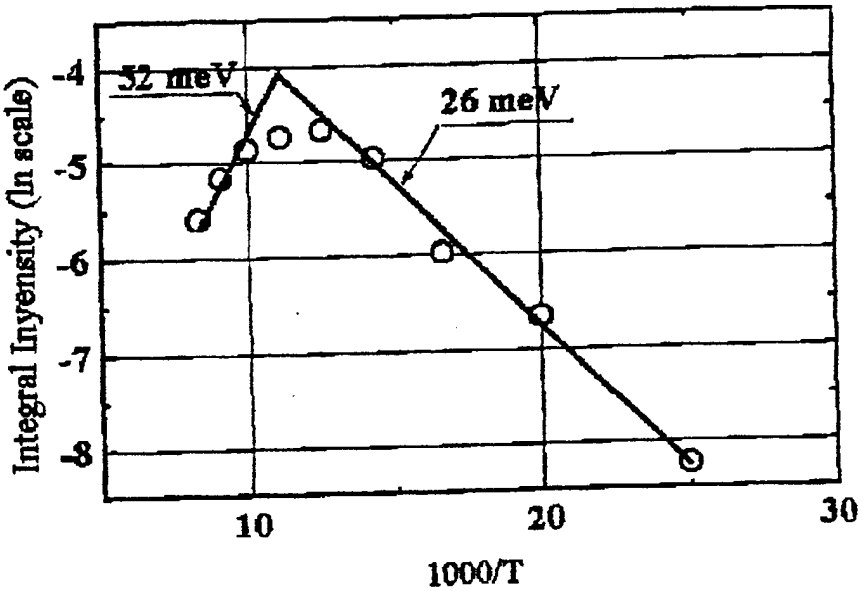

Fig.7. Temperature dependencies of the integral intensities of ESR spectra of nitrogen in cubic sites in bulk 6H SiC with $(N_D - N_A) \approx 1 \cdot 10^{16}$ cm^{-3}. The solid lines are fitted by exp(Δ/kT) with Δ = 52 meV and 26 meV.

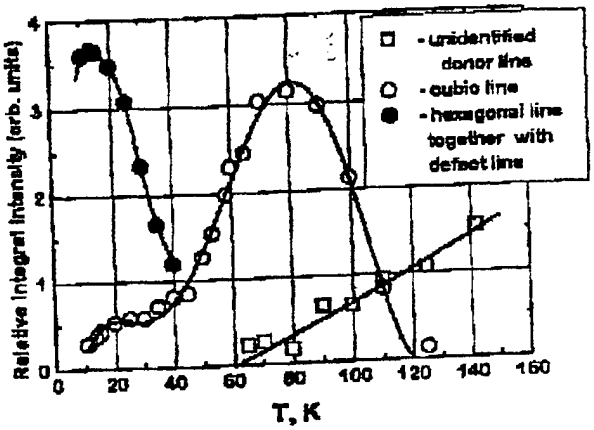

Fig.8. Temperature dependencies of the relative integral intensities of ESR spectra of 6H SiC irradiated with neutron beam (flux Φ = 2 $10^{18}cm^{-2}$, $T_{ann.}$= 1400°C). Concentration of uncompensated nitrogen (N_D-N_A) ≈ $5 \cdot 10^{17}cm^{-3}$. ν_{ESR} = 9 GHz.

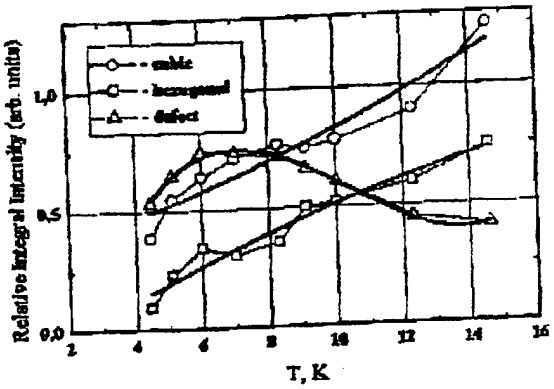

Fig. 9. Temperature dependencies of the relative integral intensities of ESR spectra of 6H SiC irradiated with neutron beam (flux Φ = $2 \cdot 10^{18}cm^{-2}$, $T_{ann.}$= 1400°C). Concentration of uncompensated nitrogen (N_D-N_A) ≈ $5 \cdot 10^{17}cm^{-3}$, ν_{ESR} =140 GHz.

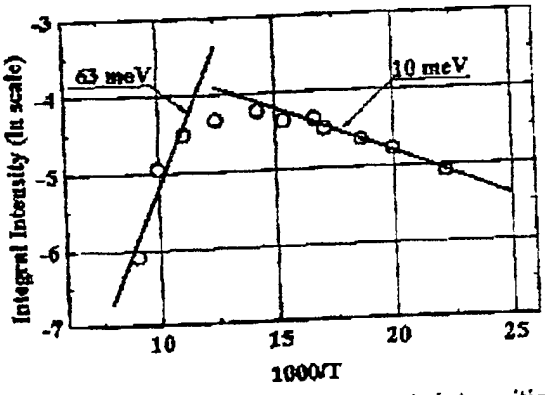

Fig.10.Temperature dependencies of the integral intensities of ESR spectra of nitrogen in cubic sites in 6H SiC irradiated with neutron beam (flux $\Phi = 2 \cdot 10^{18}cm^{-2}$, $T_{ann.}$= 1400°C). Concentration of uncompensated nitrogen ($N_D - N_A$) ≈ $5 \cdot 10^{17}cm^{-3}$. The solid lines are fitted by exp(Δ/kT) with Δ = 63 meV and 10 meV.

THE LIST of SAMPLES

#	Index	Side	Thick ness	Poly-type	Type	Impuri-ty	Concent ration	Growth direct.	Temp.
1.	MP 181.5	big	13	6H	n	Si	$6\ 10^{16}$	Si	1750
2.	MP 198.5	big	20	6H	n	Si	$8\ 10^{16}$	Si	1750
3.	89 K2		200	6H	p	Al	$5\ 10^{18}$		2000
4.	132 K3			4H	p	Al	10^{19}		2000
5.	103 K2		600	4H	n	N	$6\ 10^{17}$		2000
6.	87 K						$6\ 10^{17}$		2000
7.	126K/1			4H	n	N	$5\ 10^{16}$		2000
8.	126 K/2			4H	n	N	$5\ 10^{16}$		2000
9.	T318.2		120	6H	n	Mn			2400
10	AM 1006.4	big	25	6H	n	Si	$5\ 10^{16}$	Si	1800
11	I84		400	6H	n		$1\ 10^{16}$	Si	1900

Samples 1, 2, 10 (see fig.11, point 10 in report) native defects due to the deviation from stoichiometry.

Samples 5 - 8 bulk 4H SiC, native defect and nitrogen, behavior of exchange line (additional level?).

Sample 9 - red luminescence 1.9 eV.

Sample 11 was investigated by EPR, see fig.7, point 8 in report.

All samples were grown by sublimation sandwich method (SSM).

Principal Investigator Dr. E.N. Kalabukhova

May, 28, 1997

Letter to Sparkasse Paderborn.

To: Sparkasse Paderborn, GH-Universitat,
Warburger Str. 100, Germany.
From: Holder of Account 16110421 : Kalaboukhova Ekaterina.
Subject: Deposit of payment check
Date: April, 24, 1997.

Please deposit the payment check to my account number 16110421 of the Project SPC-97-4051 funded by Wright Air Force Lab. of materials of USA.

Yours sincerely

E.N.Kalaboukhova

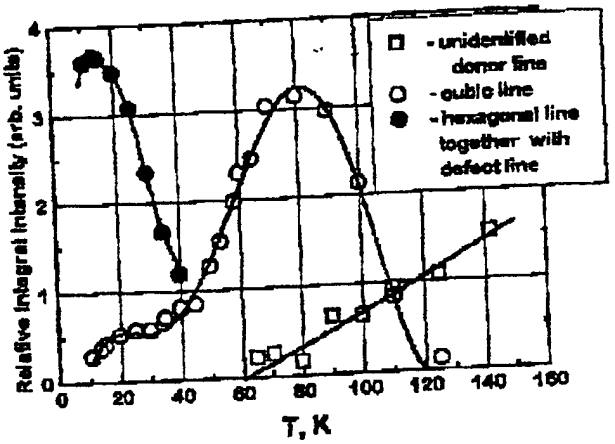

Fig.8. Temperature dependencies of the relative integral intensities of ESR spectra of 6H SiC irradiated with neutron beam (flux Φ = 2 $10^{18}cm^{-2}$, $T_{ann.}$= 1400°C). Concentration of uncompensated nitrogen $(N_D-N_A) \approx 5 \cdot 10^{17}cm^{-3}$. ν_{ESR} = 9 GHz.

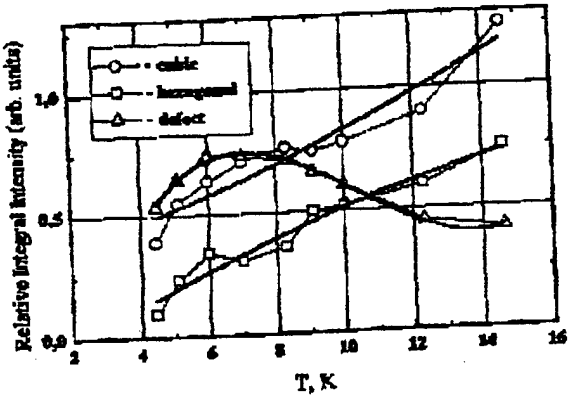

Fig. 9. Temperature dependencies of the relative integral intensities of ESR spectra of 6H SIC irradiated with neutron beam (flux $\Phi = 2 \cdot 10^{18}cm^{-2}$, $T_{ann.}$= 1400°C). Concentration of uncompensated nitrogen $(N_D-N_A) \approx 5 \cdot 10^{17}cm^{-3}$. ν_{ESR} =140 GHz.

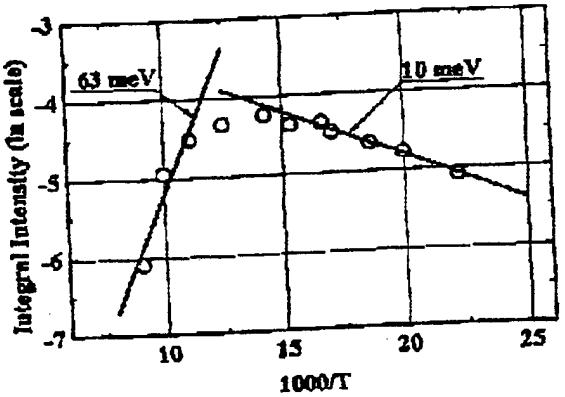

Fig.10.Temperature dependencies of the integral intensities of ESR spectra of nitrogen in cubic sites in 6H SiC irradiated with neutron beam (flux $\Phi = 2 \cdot 10^{18}cm^{-2}$, $T_{ann.}$= 1400°C). Concentration of uncompensated nitrogen $(N_D - N_A) \approx 5 \cdot 10^{17}cm^{-3}$. The solid lines are fitted by exp(Δ/kT) with Δ = 63 meV and 10 meV.

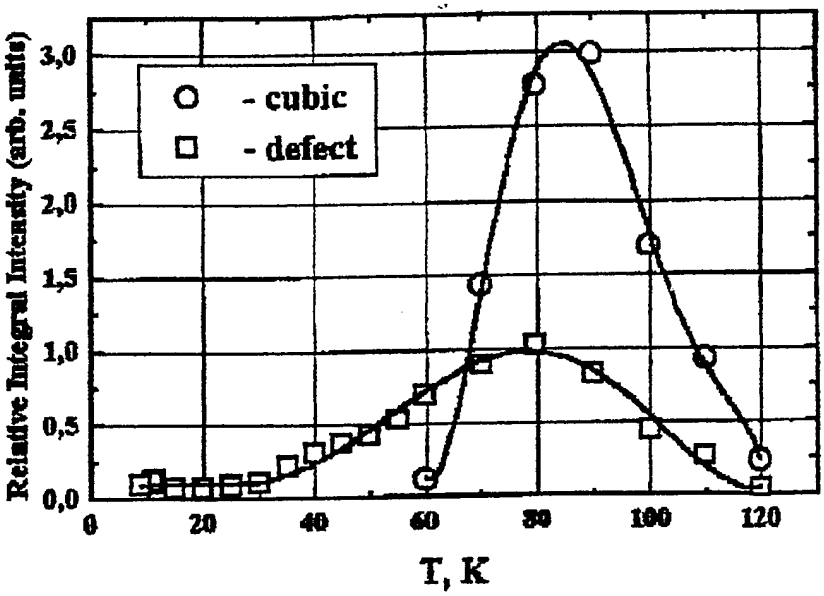

Fig.11 Temperature dependencies of the relative integral intensities of ESR spectra of 6H SiC EL with uncompensated nitrogen concentration of $(N_D - N_A) \approx 7 \cdot 10^{16} cm^{-3}$.

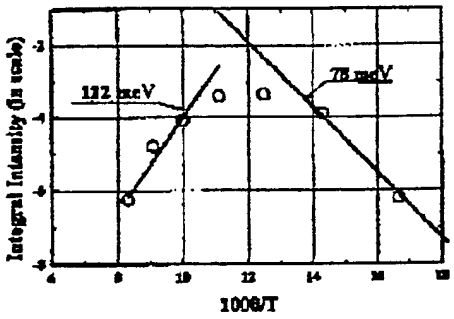

Fig.12. Temperature dependencies of the integral intensities of ESR spectra of nitrogen in cubic sites in 6H SiC EL with uncompensated nitrogen concentration of $(N_D - N_A) \approx 7 \cdot 10^{16} cm^{-3}$. The solid lines are fitted by $\exp(\Delta/kT)$ with Δ = 112 meV and 78 meV.

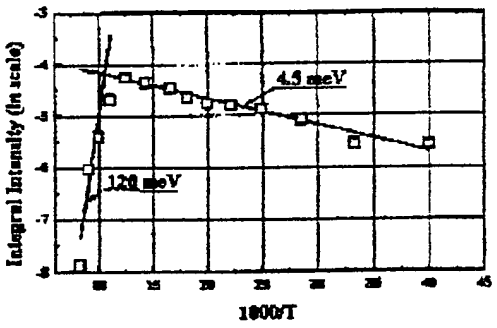

Fig.13. Temperature dependencies of the integral intensities of ESR spectra of nonidentified defect center in 6H SiC EL with uncompensated nitrogen concentration of $(N_D - N_A) \approx 7 \cdot 10^{16} cm^{-3}$. The solid lines are fitted by $\exp(\Delta/kT)$ with Δ = 120 meV and 4.5 meV.